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FINAL REPORT

ON

WASH WATER WASTE PRETREATMENT SYSTEM STUDY

Contract NAS 9-14518

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SUMMARY

The use of "real" wash water had no adverse effect on soap removal when an Olive Leaf Soap based system was used; 96 percent of the soap was removed using ferric chloride.

The Millipore plug test was evaluated and did not appear suitable for small-scale laboratory evaluations.

Numerous chemical agents were evaluated as antifoams for synthetic wash water. Wash water surfactants used included Olive Leaf Soap, Ivory Soap, Neutrogena and Neutrogena Rain Bath Gel, Alipal CO-436, Aerosol 18, Miranol JEM, Palmeto, and Aerosol MA-80. For each type of soapy wash water evaluated we were able to identify at least one antifoam capable of causing "nonpersistent" foam. In general, the silicones and the heavy metal ions (i.e., ferric, aluminum, etc.) were the most effective antifoams. Required dosage was in the range of 50 to 200 ppm.

Preliminary chemical precipitation work was begun using wash waters based on Ivory Soap, Palmeto, and Igepon CN-42. A dosage of 170 to 185 ppm ferric chloride removes 95 to 97 percent of the Ivory Soap from the wash water, while approximately 95 percent of the soap is removed from a Palmeto based wash water with a dosage of 175 to 180 ppm ferric chloride. Results with Igepon CN-42 were not promising.

Several of the more promising antifoams were evaluated for their effect on chemical precipitation of Palmeto, Olive Leaf, and Ivory Soap. Although the optimum dosage of ferric chloride coagulant needed was altered in some cases, there was no adverse effect on soap removal.

For sponge bathing and hand washing, a low-foaming liquid soap is needed. Both Triton CF-32 and blends of Pluronic L64 and L61 have low foaming characteristics by our evaluation and are worth evaluation in a formulated liquid cleanser.

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I. INTRODUCTION

Over the past decade, reverse osmosis has emerged as a convenient and efficient technique for purification of brackish and waste waters. The RO systems are generally compact, the energy requirements are relatively low since the water is not forced through a phase change, and with proper system design it is possible to obtain potable water (i.e., less than 500 ppm dissolved solids) in a single pass.

With increasingly long space flights taking place and with the possibility of orbiting space stations, it has become necessary for NASA to develop techniques to conserve and reclaim water. Perhaps the single greatest source of contaminated water from such missions is wash water from bathing and clothes washing. A typical wash water might contain approximately 0.10% detergent or soap and 0.05% NaCl; lesser amounts of urea, lactic acid, and phosphate builders; and trace amounts of miscellaneous suspended and colloidal materials such as lint, viruses, bacteria, grease, and soil. It is only natural that NASA is considering membrane separation as a basis for such a development.

Unfortunately, most of the membranes currently available have been designed primarily for salt rejection, and their operational life is adversely affected by wash water components such as detergents, bacteria, soaps, and divalent metal compounds as well as the 165°F pasturization temperature that is sometimes employed. If the objectionable constituents could be removed by a pretreatment scheme before the wash water passed through the membrane, the membrane's operation and durability would be enhanced.

Under NAS 9-13536, DeBell & Richardson, Inc., studied various techniques for eliminating objectionable wash water waste constituents. It was determined by this study that removal of objectionable constituents by a pretreatment scheme is a feasible approach. Laboratory scale tests demonstrated successful pretreatment schemes for chemical precipitation, filtration, and adsorption. These results were obtained using a simulated wash water containing Olive Leaf Soap.

Testing of the developed technique with state-of-the-art membranes and further refinement of the pretreatment processes are required. Coordination between membrane development efforts and pretreatment is to be continued. Other items being given consideration are:

a. Selection of optimum cleansing agents – which includes evaluation of human acceptability, dermatological effects, cleansing capability, compatibility with reverse osmosis membranes, lack of foaming, and susceptibility to a successful pretreatment scheme.

b. Identification and evaluation of antifoam agents for wash water based on candidate cleansing agents.

It is the purpose of the present study to provide for continued development of a wash water pretreatment scheme, resulting in an optimum concept for removing objectionable materials from spacecraft wash water waste prior to its introduction into a reverse osmosis membrane system (currently under development by NASA).

II. VERIFICATION OF PRETREATMENT RESULTS USING REAL WASH WATER

Much of the early pretreatment system development work done at D&R was based on the assumption that results with simulated wash water would be duplicated when a real wash water was used. The purpose of this portion of the effort was to verify previously promising results using a real wash water.

Under Contract NAS 9-13536 it was found that ferric chloride could be used successfully to precipitate Olive Leaf Soap from a synthetic wash water. To verify these results, we used a real Olive Leaf Soap based shower water.

The wash water was generated as follows:

(1) Twenty-eight liters of distilled water were preheated to 45°C in glass containers.

(2) Olive Leaf Soap was preweighed into a screw-cap jar (0.1% on the water).

(3) A bath tub was scoured with cleanser, rinsed three times with tap water, twice with deionized water, and then wiped dry.

(4) One of the D&R personnel jogged 2 miles in order to generate natural body oils, salts, etc.

(5) Twenty-four liters of the distilled water was placed in the tub; the remaining four liters was saved for rinsing.

(6) The subject bathed in the distilled water using all of the preweighed soap.

(7) Most of the 28 liters was recovered for precipitation and analysis.

The first step of the verification work was to reestablish the optimum dosage of ferric chloride. Based on work conducted under Contract NAS 9-13536, the optimum dosage of ferric chloride for an Olive Leaf

Soap based wash water (0.1% soap) is 170-190 ppm. A series of coagulation and flocculation experiments was conducted on the "real" water using a Hach Floc Tester and dasher mixer. The general procedure is described in Section V of this report - Preliminary Evaluation of Removal Agents and Processes. The results appear in Table 1.

A dosage of 200 ppm ferric chloride removes almost 95 percent of the Olive Leaf Soap.

TABLE 1

Soap Removal from Real Olive Leaf Soap Based Wash Water(1)

Experiment Number	Coagulant/Flocculant (ppm)		pH		(2) Floccula-tion Size and Rate	(3) Settling Rate	Actual Soap Content (ppm)	% Soap Removed
	$FeCl_3$	Reten 425	Initial	Final				
A-3313-A	170	-	8.0	4.3	1	2	-	-
A-3313-B	180	-	8.0	4.2	2	2	-	-
A-3313-C	190	-	8.0	3.7	5	4	73	93.5
A-3313-D	200	-	8.0	3.7	5	4	59	94.8
A-3313-E	210	-	8.0	3.6	1	2	-	-
A-3313-F	220	-	8.0	3.5	1	2	-	-
A-3313-G	190	-	8.0	3.8	5	4	74	93.4
A-3313-H	190	1.0	8.0	3.8	5	4	73	93.5
A-3313-I	200	1.0	8.0	3.6	2	2	-	-

(1) Carbon tetrachloride/IR analysis for carbonyl indicates an actual soap content of 1125 ppm rather than the usual 1000.

(2) Flocculion – rated on a scale of 0-5:

0 - no floc
1 - slow growth; small floc
2 - slow growth; larger floc than (1)
3 - faster growth
4 - large floc in less than 1 minute
5 - large floc almost immediately

(3) Settling – rated on a scale of 0-5:

0 - no floc
1 - no settling
2 - very little settling (less than 10%)
3 - 99-100% settled out in 20 minutes
4 - 99-100% settled out in 2-3 minutes
5 - settles out in less than 2 minutes

III. EVALUATION OF MILLIPORE SCREENING PROCEDURE

The purpose of this task was to evaluate the Millipore Screening procedure as a method for determining the efficiency of pretreatment concepts.

The procedure, used by du Pont during a recent OSW program, is described in a progress report as follows:

> "A more reliable and meaningful term to express the solids level in the pretreated seawater supplied to the RO systems is the plug factor. The change in rate of filtration through a 0.45-micron Millipore filter at a standard pressure of 30 psi over a fixed time interval is a direct measure of the accumulation of particulate matter on the filter – i.e., of the solids content of the water used for the test. The standard time interval for clear water is 15 minutes but is preferably shortened if the water has a relatively high solids content. Results are expressed as percentage drop in filtrate flow as in the example
>
> $$P^{15}_{30} = 50\%$$
>
> where the superscript identifies the time interval (minutes) and the subscript expresses the applied pressure (psi). The theoretical boundaries are 0% – i.e., water free of any interfering matter – and 100%, which means water that does not yield any filtrate at the applied pressure."

To run the plug test we used Millipore HAWP090 filters (0.45-micron nominal pore diameter) fitted in a brass pressure filter funnel (4.5 cm diameter, 130 cc capacity). The funnel was pressurized from the top with nitrogen.

In testing the system, we used distilled water. At 30 psig, the filter funnel emptied itself in a few seconds.

To provide greater capacity, a 2-liter, stainless steel reservoir was then attached to the top of the filter funnel and nitrogen pressure was applied

from the top. The plug test on distilled water was repeated at 10 psig. The results for five determinations are:

Flow Rate (cc/min.) At 0 Time	At 5 Minutes	P_{10}^{5} (%)
246	102	59
348	57	84
366	66	82
480	189	61
495	114	77

Each trial was done with a fresh filter. Water, being a very polar fluid, is prone to contamination; therefore, the decline in water flow rate is rather severe. Agreement between tirals here is poor.

The plug test was repeated with untreated Olive Leaf Soap based wash water (0.1% soap). Results were as follows:

Flow Rate (cc/min.) At 0 Time	At 5 Minutes	P_{10}^{5} (%)
69	0.4	99.4
51	0.3	99.4

The filters were plugged quickly, presumably by colloidal soap.

We then coagulated and flocculated 2 liters of the wash water with 180 ppm ferric chloride, prefiltered the water through Whatman Number 1 paper, and ran the plug test. The experiment was done in duplicate and in both cases the initial flow rate was approximately 590 cc/min. and the reservoir emptied in less than 4 minutes, preventing a second reading.

When we used water coagulated with excess ferric chloride (200 ppm), the initial flow rate during the plug test was still 590 cc/min., and again the reservoir emptied before a second flow rate (5 minutes) could be taken.

We feel that the plug test was unsatisfactory as a screening technique for the following reasons:

(1) It appeared that the test is designed for pilot scale work. If the test were conducted as described (i.e., flow drop over 15 minutes at 30 psig), several gallons of water would be needed.

(2) Even when conducted at reduced pressure and for shorter time, the test required fairly large quantities of water, which is inconvenient for lab evaluation work.

(3) Prefiltering of large quantities of coagulated wash water tends to be time consuming. Two hours were required, for example, to prefilter 2 liters of wash water which had been coagulated with 200 ppm ferric chloride.

(4) Even at reduced pressure and time, the plug test often exceeded the capacity of our laboratory equipment.

For these reasons, no further work was done with the plug test.

IV. PRELIMINARY SELECTION AND EVALUATION OF ANTIFOAM AGENTS

As part of the wash water recovery, it will be necessary to prevent or eliminate the foams that are often encountered with agitated soapy water. One method of eliminating foams is through the use of antifoam agents.

We contacted several antifoam suppliers for candidate materials; the following conventional antifoams were evaluated:

Material	Manufacturer	Type
Nalco 73C44 Nalco 73C37	Nalco Chemical Co.	Amides
Nalco 71-D5, 8616, 867, and 4WP126	Nalco Chemical Co.	Fatty acid esters
Dow Corning DB-110 Antifoam 71, AF-75	Dow Corning Corp. General Electric	Silicones
Foamaster, Foamaster B, VL, AP, G, and TMC	Nopco Chemical Div., Diamond-Shamrock	Proprietaries
Foamex/Pegosperse 100L	Glyco Chemical	
Colloid 680 and 681F	Colloids, Inc.	
Nalco 121	Nalco Chemical Co.	

The fatty acid esters were supplied as solutions in either isopropanol or kerosene and therefore were not evaluated. Nalco 73C37 and Foamaster G were not compatible with water.

In addition to conventional antifoams, ferric chloride, calcium chloride, aluminum sulfate and magnesium sulfate were screened.

All antifoam evaluations were done on synthetic wash water based on the following formulation:

Material	Concentration (ppm)
Surfactant (soap)	1000[a]
Sodium chloride	500
Sodium sulfate	150
Lactic acid	100
Urea	50

(a) Solids or active soap

Antifoam agents were added from 1 percent aqueous dispersions.

The procedure for the foaming test is as follows:

(1) Weigh the candidate antifoam into a clean 250 ml graduated cylinder.

(2) Add 100 ml of the appropriate synthetic wash water.

(3) Shake the cylinder for 15 seconds.

(4) Place the cylinder on the bench, and measure the foam height above the liquid in centimeters after 15 and 60 seconds.

Antifoams were screened initially at 200 ppm; promising antifoams were also evaluated at 100 and 50 ppm.

The first set of antifoam studies was done with an Olive Leaf Soap based wash water (Table 2). Olive Leaf Soap is a moderate foamer, giving 10 cm of foam at 15 seconds by the above shake test (no antifoam). Of the conventional antifoams evaluated, only the silicones were effective; both DB-110 and Antifoam 71 gave nonpersistent foams at 100 ppm. Antifoam AF-75 destroyed the foam at 150 ppm.

Heavy metal ions also gave satisfactory results; they presumably precipitate a portion of the soap which forms metal palmitates and stearates, which act as antifoams. Aluminum sulfate gave a nonpersistent foam at 200 ppm, while ferric chloride was effective at 100 ppm.

Ivory soap is a high foamer; a shake test on the wash water control (no antifoam) gave a 15-second foam height of greater than 18 cm (Table 3). Of the conventional antifoams, both DB-110 and Antifoam 71 were satisfactory. Ferric chloride and calcium chloride were also effective in eliminating the foam.

Neutrogena is a surprisingly low foamer; our shake test indicated a 15-second foam height of only 1.5 cm for the Neutrogena soap based wash water. Several of the antifoams evaluated were effective in reducing foam (Table 4).

Aerosol MA-80, sodium dihexyl sulfosuccinate, is a moderate foamer; a shake test on a wash water control (no antifoam) gave 8 to 10 cm of foam in 15 seconds (Table 5). The most effective antifoam investigated for MA-80 was ferric chloride, which gave "nonpersistent" foam with a dosage as low as 50 ppm. Several of the conventional antifoam agents also gave significant foam reductions.

Alipal CO-436, the sodium salt of a sulfated alkylphenoxy-poly(oxyethylene) ethanol, is a very high foamer; our shake test indicated a 15-second foam height in excess of 25 cm. The only effective antifoam for Alipal CO-436 among the ones evaluated is a silicone, Antifoam 71 (Table 6).

Aerosol 18 is a moderate foamer, giving 12-15 cm of foam at 15 seconds by the standard shake test (no antifoam) (Table 7). Of the antifoams evaluated, only AF-75 and DB-110 came close to being effective. Both allowed 0.1 to 0.2 cm of foam in 15 seconds at a dosage of 200 ppm.

Miranol JEM is a low foamer at this concentration (0.1% in a synthetic wash water); a shake test on the wash water control (no antifoam) gave a 15-second foam height of only 1.0 to 1.4 cm (Table 8). Of the antifoams evaluated, only the silicones were effective in eliminating foam. A dosage of only 50 ppm of either antifoam emulsion AF-75 or DB-110 resulted in "nonpersistent" foam after 15 seconds.

Neutrogena Rain Bath Gel is a high foamer; a shake test on a wash water control gave 27-28 cm of foam in 15 seconds (Table 9). As with Miranol JEM, the only effective antifoam agents were the silicones, but the required dosages were large. Antifoam DB-110 was effective at 300 ppm, while AF-75 was borderline at 500 ppm.

Rochester Germicide has made a product change. Olive Leaf Soap has been discontinued and has been replaced by Palmeto. The new material is reported to have 19% solids including an emollient to prevent "dryness". By our test, the soap contains 19.1% solids.

To help verify the performance of Palmeto, we ran a series of antifoam experiments (Table 10). Palmeto is a much lower foamer than Olive Leaf (3 cm versus 10 cm, respectively, after 15 seconds); this is probably due to the presence of the emollient, perhaps an excess of fatty acid, which helps suppress the foam.

Of the commercial antifoams evaluated, only the silicones proved effective, allowing only 0.1 cm of foam at 200 ppm. The new candidate, SWS-211, is a fine emulsion silicone. Ferric chloride was also effective, but only at 100 ppm; higher dosages produced a foamy iron/fatty acid sludge.

From our work so fat, it appears that there will be no difficulty in finding a satisfactory antifoam for each candidate soap.

CONCENTRATED SYNTHETIC WASH WATER

Another component in the overall water reclamation systems which may have a foaming problem is the VCD (vapor compression distillation unit). These units will undoubtedly have to handle the concentrated soapy reject waters from RO and/or ultrafiltration. Soap strengths in these concentrates could run in excess of 1%; such high concentrations of soap could cause a foaming problem during distillation.

To check the feasibility of using antifoam agents to combat foam in concentrated soap solutions, we employed the same shake test as was used with dilute wash waters. The procedures and materials were the same except for the synthetic wash water, which contained the following:

Material	Concentration ppm	Concentration %
Olive Leaf Soap (active solids)	10,000	1.0
Lactic acid	1,000	0.1
Urea	500	0.05
NaCl	5,000	0.5
$Na_2(SO_4)$	1,500	0.15

The antifoams used were only those agents which showed promise with dilute wash water. The results are presented in Table 11. At room temperature, DB-110 was reasonably effective at 1000 ppm (0.1%). At 40°C, both AF-75 and DB-110 were effective at 1250 ppm. Ferric chloride gave confusing results. In all cases it coagulated the soap and produced a foam floc which floated to the surface of the wash water

TABLE 2

Effect of Antifoam Agents on Foam Stability – Olive Leaf Soap Based Wash Water

Antifoam Agent	Manufacturer	Residual Foam Height At Various Antifoam Dosages (ppm) (1)							
		50 ppm		100 ppm		150 ppm		200 ppm	
		15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Foamex/ Pegosperse 100L (3:1)	Glyco Chemical	- -	- -	7.5 8.0	7.0 7.2	- -	- -	7.5 4.5	~7 4.1
Foamaster AP	Nopco Chemical	- -	- -	3.5 4.2	3.5 4.0	- -	- -	3.1 3.3	3.1 3.2
Foamaster B	Nopco Chemical	- -	- -	6.5 6.0	6.0 5.5	- -	- -	- -	- -
Foamaster VL	Nopco Chemical	- -	- -	3.7 3.7	3.5 3.7	- -	- -	4.1 4.1	3.8 3.9
73C44	Nalco Chemical	- -	- -	3.2 3.4	3.2 3.2	- -	- -	2.9 3.5	2.8 2.0
Foamaster	Nopco Chemical	-	-	-	-	-	-	4.2	2.8
Foamaster TMC	Nopco Chemical	-	-	-	-	-	-	4.0	3.2
121	Nalco Chemical	-	-	-	-	-	-	2.8	2.5
Colloid 680	Colloids, Inc.	-	-	-	-	-	-	2.4	2.4
Colloid 681F	Colloids, Inc.	-	-	-	-	-	-	2.2	1.7

(1) NASA Shake Test; measures height of foam in cm in a graduated cylinder, 15 seconds after shaking.

...Continued

Table 2 (Continued - 2)

Antifoam Agent	Manu-facturer	Residual Foam Height at Various Antifoam Dosages (ppm)							
		50 ppm		100 ppm		150 ppm		200 ppm	
		15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Antifoam 71	General Electric	0.3 -	0.1 -	0 0	- -	- -	- -	0 0	0 0
AF-75	General Electric	- -	- -	1.0 1.0	0.1 0.1	0 0	0 0	0 0	0 0
DB-110	Dow Corning	0.8 1.4	- -	0 0	0 0	- -	- -	0 0	0 0
Ferric Chloride	-	0.4 0.4	- 0.3	0 0	0 0	- -	- -	- -	- -
Calcium Chloride	-	- -	- -	0.3 0.3	0 0	0.6 -	0 -	0.4 -	- -
Aluminum Sulfate	-	- -	- -	0.7 -	0.6 -	0.2 0.2	0 0	0 0	0 0

Control (no antifoam):

Seconds	Foam Height
15	10/9.5
60	10/9.5

TABLE 3

Effect of Antifoam Agents on Foam Stability - Ivory Soap Based Wash Water

Anti-foam Agent	Manufacturer	Residual Foam Height (cm) at Various Antifoam Dosages (ppm)								
		50 ppm			100 ppm			200 ppm		
		15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
Magnesium Sulfate	-	2.8 2.5	2.5 1.2	2.0 1.0	2.5 2.3	1.8 1.9	1.5 1.7	1.8 1.5	1.5 1.3	1.2 1.0
Calcium Chloride	-	5.0 3.6	5.0 3.6	5.0 3.3	0.5 0.8	0.2 0.7	0.2 0.6	0.1 0.1	0.1 0.1	0 0
Aluminum Sulfate	-	- -	- -	- -	- -	- -	- -	2.0 2.0	1.7 1.5	1.5 1.2
Ferric Chloride	-	0.6 0.5	0.5 0.4	0.4 0.3	0.3 0.5	0.3 0.4	0.2 0.3	0.2 0	0 0	0 0
Foamaster	Nopco Chemical	- -	- -	- -	>16 >16	8.8 7.3	8.5 7.0	5.5 5.8	5.0 5.8	5.0 5.4
Foamaster TMC	Nopco Chemical	- -	- -	- -	>17 >16	9.5 9.5	9.3 9.2	>16 >16	8.7 8.8	7.9 8.4
Foamaster AP	Nopco Chemical	- -	- -	- -	- -	- -	- -	5.5 6.0	5.4 5.8	5.3 5.6
Colloid 680	Colloids, Inc.	>15 >15	7.2 5.9	7.0 5.5	4.4 5.1	4.3 4.9	4.2 4.8	2.7 2.4	2.5 2.0	2.0 1.8
Colloid 681 F	Colloids, Inc.	>15 >15	7.5 7.0	7.0 6.7	4.5 4.0	4.5 3.7	4.3 3.5	1.7 1.6	1.5 1.2	1.4 1.2

...Continued

Table 3 (Continued - 2)

Anti-foam Agent	Manu-facturer	Residual Foam Height (cm) at Various Antifoam Dosages (ppm)								
		50 ppm			100 ppm			200 ppm		
		15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
Foamex/ Pegosperse 100L (3:1)	Glyco Chemical	- -	- -	- -	- -	- -	- -	>15 8	9.0 7.6	8.4 7.0
73C44	Nalco	- -	- -	- -	- -	- -	- -	3.0 3.2	2.7 2.6	2.4 2.5
DB-110	Dow Corning	3.8 2.9	3.5 2.7	3.3 2.2	0.6 0.2	0.5 0.1	0.5 0.1	0.1 0.1	0.1 0.1	0 0
AF-71	General Electric	0.5 0.8	0.3 0.7	0.3 0.4	0.1 0.1	0.1 0.1	0.1 0.1	0 0	0 0	0 0
AF-75	General Electric	- -	- -	- -	- -	- -	- -	1.5 ~2	- -	1.2 ~2

Control (no antifoam):	Seconds	Foam Height (cm)
	15	>18/ > 16
	30	>18/ > 16
	60	15/ 13

TABLE 4

Effect of Antifoam Agents on Foam Stability - Neutrogena Soap Based Wash Water

Anti-foam Agent	Manu-facturer	Residual Foam Height (cm) at Various Antifoam Dosages (ppm)								
		50 ppm			100 ppm			200 ppm		
		15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
Magnesium Sulfate	-	0.1 0.1	0.1 0.1	0.1 0.1	0.4 0.3	0.3 0.2	0.2 0.1	0.2 0.2	0.1 0.2	0.1 0.2
Calcium Chloride	-	0.3 0.3	0.2 0.3	0.2 0.2	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1
Aluminum Sulfate	-	- -	- -	- -	0.5 0.3	0.3 0.2	0.2 0.2	0.2 0.4	0.1 0.3	0.1 0.2
Ferric Chloride	-	0.2 0.3	0.1 0.2	0.1 0.2	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.2	0.1 0.1	0.1 0.1
Foamaster	Nopco Chemical	0.5 0.3	0.4 0.3	0.3 0.3	0.4 0.4	0.2 0.2	0.1 0.2	0.1 0.2	0.1 0.1	0.1 0.1
Foamaster TMC	Nopco Chemical	0.5 0.7	0.5 0.3	0.4 0.3	0.3 0.3	0.2 0.2	0.2 0.2	0.3 0.3	0.3 0.3	0.1 0.1
Foamaster AP	Nopco Chemical	- -	- -	- -	- -	- -	- -	0.5 0.8	0.5 0.6	0.4 0.6
Colloid 680	Colloids, Inc.	0.3 0.4	0.2 0.4	0.2 0.3	0.3 0.2	0.2 0.2	0.2 0.2	0 0.1	0 0.1	0 0
Colloid 681 F	Colloids, Inc.	- -	- -	- -	0.4 0.2	0.3 0.2	0.2 0.1	0.3 0.2	0.2 0.1	0.2 0.1

... Continued

Table 4 (Continued - 2)

Anti-foam Agent	Manufacturer	Residual Foam Height (cm) at Various Antifoam Dosages (ppm)								
		50 ppm			100 ppm			200 ppm		
		15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
73C44	Nalco Chemical Co.	- -	- -	- -	0.2 0.2	0.2 0.2	0.1 0.1	0.1 0.1	0 0.1	0 0
DB-110	Dow Corning	0.2 0.2	0.1 0.2	0.1 0.1	0.3 0.2	0.1 0.1	0 0	0.1 0.1	0.1 0.1	0 0
AF 71	General Electric	0.2 0.2	0.1 0.2	0.1 0.1	0.1 0.2	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1	0 0

Control (no antifoam):

Seconds	Foam Height (cm)
15	1.5/1.4
30	1.5/1.4
60	1.3/1.3

TABLE 5

Effect of Antifoam Agents on Foam Stability
Aerosol MA-80 Based Wash Water

Antifoam Agent	Manufacturer	Residual Foam Heights at Various Antifoam Dosages: 50 ppm, 15 sec (cm)	50 ppm, 60 sec (cm)	100 ppm, 15 sec (cm)	100 ppm, 60 sec (cm)	200 ppm, 15 sec (cm)	200 ppm, 60 sec (cm)
Magnesium Sulfate	-	- -	- -	- -	- -	8.2 7.5	0.1 0.1
Calcium Chloride	-	- -	- -	- -	- -	4.5 5.0	0 0
Aluminum Sulfate	-	- -	- -	0.2 0.3	0 0	0.1 0	0 0
Ferric Chloride	-	0 0	0 0	0 0	0 0	0 0	0 0
Antifoam 71	General Electric	- -	- -	- -	- -	0.4 0.5	0.2 0.4
DB-110	Dow Corning	- -	- -	- -	- -	0 0	0 0
Foamaster	Nopco Chemical	- -	- -	0.2 0.2	0.1 0.1	0.1 0.1	0.1 0.1
Foamaster TMC	Nopco Chemical	- -	- -	0.2 0.2	0.1 0.2	0.1 0.1	0.1 0.1
Foamaster AP	Nopco Chemical	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0	0.1 0.1	0 0
Foamaster B	Nopco Chemical	- -	- -	- -	- -	0.3 0.3	0.3 0.3
Foamaster VL	Nopco Chemical	0.2 0.1	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1
Foamex/ Pegosperse 100L	Glyco Chemical	- -	- -	- -	- -	3.5 4.0	1.3 1.6
73C44	Nalco Chemical	0.4 0.2	0 0	0.1 0.1	0.1 0.1	0.1 0.1	0.1 0.1
Colloid 680	Colloids, Inc.	0.1 0.1	0 0	0.1 0.2	0 0.1	0.1 0.1	0 0
Colloid 681F	Colloids, Inc.	0.2 0.4	0 0	0 0.1	0 0	0 0.1	0 0

Control (no antifoam):

Seconds	Foam Height (cm)
15	8.0/10.5
30	3.0/6.0
60	0.1/0.2

TABLE 6

Effect of Antifoam Agents on Foam Stability
Alipal CO-436 Based Wash Water

Antifoam Agent	Manufacturer	Residual Foam Heights at Various Antifoam Dosages (ppm)			
		100 ppm		200 ppm	
		15 sec	60 sec	15 sec.	60 sec
Magnesium Sulfate	-	- -	- -	26 24	23 21
Calcium Chloride	-	- -	- -	25 25	21 21
Aluminum Sulfate	-	- -	- -	25 24	21 21
Ferric Chloride	-	- -	- -	20 20	17 16
Foamaster	Nopco Chemical	- -	- -	16 12	11 10
Foamaster TMC	Nopco Chemical	- -	- -	14 14	12 12
Foamaster AP	Nopco Chemical	- -	- -	15 15	6.8 7.9
Foamaster B	Nopco Chemical	- -	- -	9.0 9.4	8.1 8.3
Foamaster VL	Nopco Chemical	- -	- -	6.7 8.6	5.5 7.7
DB-110	Dow Corning	- -	- -	0.4 0.4	0.2 0.3
Antifoam 71	General Electric	0.5 0.6	0.3 0.3	0.1 0.1	0.1 0.1
Foamex/ Pegosperse 100L	Glyco Chemical	- -	- -	16 17	13 12
73C44	Nalco Chemical Co.	- -	- -	3.3 2.4	2.1 2.1
Colloid 680	Colloids, Inc.	- -	- -	8.0 10.8	6.9 9.5
Colloid 681 F	Colloids, Inc.	- -	- -	2.4 1.8	1.8 1.4

Control (no antifoam):

Seconds	Foam Height (cm)
15	>25/>26
30	>20/>20
60	>20/>20

TABLE 7

Effect of Antifoam Agents on Foam Stability - Aerosol 18 Based Wash Water

Antifoam Agent	Manufacturer	Residual Foam Height (cm) at Various Antifoam Dosages			
		100 ppm		200 ppm	
		15 sec	60 sec	15 sec	60 sec
Colloid 681F	Colloids, Inc.	- -	- -	2.3 2.5	2.2 2.3
Colloid 680	Colloids, Inc.	- -	- -	2.6 2.1	2.4 2.0
Foamaster AP	Nopco Chemical Div., Diamond Shamrock Chem.	- -	- -	>6 >5	>5 4.2
Foamaster TMC	Same	- -	- -	>2.5 2.8	2.0 2.0
Foamaster VL	Same	- -	- -	>3 ~5	2.2 2.8
Foamaster B	Same	- -	- -	2.7 ~4	2.4 3.7
AF-75	General Electric	0.6 0.4	0.5 0.4	0.1 0.2	0.1 0.2
DB-110	Dow Corning	- -	- -	0.2 0.1	0.2 0.1
73 C44	Nalco Chemical	- -	- -	2.0 1.7	1.8 1.6
3:1 Foamex/ Pegosperse 100 L	Glyco Chemical	- -	- -	>5 ~6	>5 ~6
Calcium chloride	-	- -	- -	~10 >9	~9 ~9
Ferric chloride	-	- -	- -	2.7 1.8	2.7 1.8

Control (no antifoam):	Seconds	Foam Height (cm)
	15	13/12
	30	12.8/>11
	60	11.8/>10

TABLE 8

Effect of Antifoam Agents on Foam Stability - Miranol JEM Based Wash Water

Antifoam Agent	Manufacturer	Residual Foam Height (cm) at Various Antifoam Dosages			
		100 ppm		200 ppm	
		15 sec	60 sec	15 sec	60 sec
Foamaster B	Nopco Chemical Div., Diamond Shamrock Chem.	- -	- -	0.4 0.5	0.4 0.4
Foamaster TMC	Same	- -	- -	0.6 0.7	0.4 0.4
Foamaster VL	Same	- -	- -	0.8 0.8	0.7 0.7
Foamaster	Same	- -	- -	0.6 0.6	0.5 0.5
Foamaster AP	Same	- -	- -	1.3 0.9	0.8 0.8
Antifoam 71	General Electric	0.1 0.1	0 0	0 0.1	0 0
Antifoam AF-75	Same	0 0	0 0	50 ppm 0 0	0 0
DB-110	Dow Corning	0 0	0 0	50 ppm 0 0	0 0
3:1 Foamex/ Pegosperse 100 L	Glyco Chemical	- -	- -	0.8 0.9	0.5 0.8
Colloid 681F	Colloids, Inc.	- -	- -	0.4 0.4	0.4 0.4
Colloid 680	Same	- -	- -	0.4 0.4	0.3 0.3

. . . Continued

Table 8 (Continued - 2)

Antifoam Agent	Manufacturer	Residual Foam Height (cm) at Various Antifoam Dosages			
		100 ppm		200 ppm	
		15 sec	60 sec	15 sec	60 sec
73 C44	Nalco Chemical	- -	- -	0.8 0.8	0.6 0.6
Ferric chloride	-	- -	- -	>8 -	>8 -
Calcium chloride	-	- -	- -	1.5 1.4	1.1 1.1
Magnesium Sulfate	-	- -	- -	1.5 1.4	1.3 1.2

Control (no antifoam):	Seconds	Foam Height (cm)
	15	1.0/1.4
	30	0.9/1.2
	60	0.9/1.0

TABLE 9

Effect of Antifoam Agents on Foam Stability - Neutrogena Rain Bath Gel Based Wash Water

Antifoam Agent	Residual Foam Height (cm) at Various Antifoam Dosages							
	100 ppm		200 ppm		300 ppm		500 ppm	
	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Foamaster	- -	- -	- -	- -	- -	- -	4.3 4.5	4.2 4.2
Foamaster TMC	- -	- -	- -	- -	- -	- -	10 8	8.4 7.7
Foamaster AP	- -	- -	- -	- -	- -	- -	3.5 3.0	3.2 2.6
Foamaster B	- -	- -	- -	- -	- -	- -	~ 5 3.8	4.6 3.6
Foamaster VL	- -	- -	- -	- -	- -	- -	4.4 4.1	4.0 3.4
DB-110	4.0 4.0	3.1 3.2	0.1 0.1	< 0.1 < 0.1	< 0.1 0	0 0	- -	- -
AF-75	- -	- -	0.3 0.4	0.2 0.3	0.1 0.1	0.1 0.1	< 0.1 < 0.1	< 0.1 < 0.1
Ferric chloride	- -	- -	19 20	16 16	- -	- -	13 13	10.5 11.0
Calcium chloride	- -	- -	- -	- -	- -	- -	25 25	23 21.5
Aluminum sulfate	- -	- -	> 21 > 22	19 19	- -	- -	19.5 > 18.0	15.7 15.1
Magnesium Sulfate	- -	- -	- -	- -	- -	- -	25 > 24	22.3 21

...Continued

Table 9 (Continued - 2)

Antifoam Agent	Residual Foam Height (cm) at Various Antifoam Dosages							
	100 ppm		200 ppm		300 ppm		500 ppm	
	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Colloid 680	- -	- -	- -	- -	- -	- -	5.0 3.5	4.5 3.3
Colloid 681F	- -	- -	- -	- -	- -	- -	2.9 3.2	2.9 2.9
73 C44	- -	- -	- -	- -	- -	- -	1.3 1.2	1.3 1.0
3:1 Foamex/ Pegosperse 100 L	- -	- -	- -	- -	- -	- -	>12 >15	11.0 11.2

Control (no antifoam): **Height of Foam (cm)**

15 sec	28/27
30 sec	26/25
60 sec	24/24

TABLE 10

Effect of Antifoam Agents on Foam Stability - Palmeto Based Wash Water

Antifoam Agent	Residual Foam Height (cm) at Various Antifoam Dosages							
	100 ppm		150 ppm		200 ppm		250 ppm	
	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Ferric chloride	~0 0.1	~0 0.1	0.7 0.8	0.7 0.8	1.5 * -	1.5 * -	- -	- -
Aluminum sulfate	0.8 0.8	0.7 0.7	- -	- -	0.3 * 0.3 *	0.3 * 0.2 *	- -	- -
Magnesium sulfate	- -	- -	- -	- -	3.3 3.4	1.9 2.0	- -	- -
Calcium chloride	- -	- -	- -	- -	0.9 0.5	0.5 0.4	- -	- -
Antifoam AF-75	1.0 -	0.5 -	- -	- -	0.1 0.1	0.1 0.1	0.8 0.7	0.1 0.1
DB-110	1.5 -	0.5 -	- -	- -	0.1 -	0.1 -	0 0.1	0 0.1
SWS-211	- -	- -	- -	- -	0.1 -	0.1 -	0.1 0.1	0.1 0.1
73C44	- -	- -	- -	- -	2.5 3.0	2.4 2.6	- -	- -
Colloid 681F	- -	- -	- -	- -	2.4 2.5	1.9 2.2	- -	- -
Colloid 680	- -	- -	- -	- -	2.1 2.5	1.8 2.0	- -	- -
Foamaster TMC	- -	- -	- -	- -	2.3 2.2	2.0 1.8	- -	- -
Foamaster B	- -	- -	- -	- -	2.2 2.2	2.0 1.8	- -	- -

Control (no antifoam):

Foam Height (cm)
15 sec > 3/3/2.8
60 sec 2.2/2.2/2.5

* Forms a foamy sludge that floats to the surface.

TABLE 11

Effect of Antifoam Agents on Foam Stability - Concentrated (1% Soap) Olive Leaf Soap Based Wash Water

Antifoam Agent	Water Temp. (°C)	Residual Foam Height (cm) at Various Antifoam Dosages							
		750 ppm		1000 ppm		1250 ppm		1500 ppm	
		15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
DB-110	23	0.4 0.2	0.2 < 0.2	0.1 0.1	0.1 < 0.1	0.1 < 0.1	0.1 < 0.1	- -	- -
DB-110	40	- -	- -	0.2 0.2	0.1 0.1	< 0.1 < 0.1	< 0.1 < 0.1	- -	- -
Antifoam AF-75	23	- -	- -	0.3 0.4	0.2 0.3	0.2 0.2	0.2 0.2	- -	- -
Antifoam AF-75	40	- -	- -	0.5 0.4	0.3 0.3	0.1 0.1	< 0.1 < 0.1	< 0.1 < 0.1	0 < 0.1
Ferric chloride	23	3.7 * 3.9	3.0 * 3.3	4.0 * 4.5	4.0 * 4.0	- -	- -	5.0 * 5.5	4.8 * 5.0
Ferric chloride	40	- -	- -	- -	- -	4.4 5.0	3.9 4.4	- -	- -

Control (no antifoam):

Seconds	Foam Height (cm) 23°C	Foam Height (cm) 40°C
15	28/29	29/28
30	27/28	27/26
60	25/25	25/24

* These figures represent a porous sludge (i.e., a mixture of foam and precipitated soap) which floated to the surface.

V. PRELIMINARY EVALUATION OF REMOVAL AGENTS AND PROCESSES

Under contract NAS 9-13536, Wash Water Solids Removal System Study, we demonstrated the feasibility of chemical precipitation of Olive Leaf Soap. During the present program we continued our evaluation of removal agents for chemical precipitation. New candidate cleansing agents or surfactants were procured as follows:

Material	Type	Active Ingredients	Manufacturer
Ivory Soap	Soap-based cleansing agent	Sodium stearate	Proctor and Gamble
Igepon CN-42	Low-foaming synthetic surfactant	N-cyclohexyl N-palmitoyl taurate	GAF Corp.
Aerosol MA	Synthetic surfactant	Sodium dihexyl sulfosuccinate	American Cyanamid
Aerosol 22	Synthetic surfactant	Tetrasodium N-(1,2-dicarboxyethyl)-N-octadecyl sulfosuccinate	American Cyanamid
Aerosol 102	Synthetic surfactant	A C_{10} to C_{12} ethoxylated disodium sulfosuccinate	American Cyanamid
Aerosol 18	Synthetic surfactant	Disodium N-octadecyl sulfosuccinamate	American Cyanamid
Aerosol GPG	Synthetic surfactant	A nonyl phenoxy ethoxylated disodium sulfosuccinate	American Cyanamid

General procedures for coagulation, flocculation, and evaluation have been described in "Wash Water Solids Removal System Study", Final

Report to NASA-JSC under Contract NAS 9-13536, prepared by DeBell & Richardson. A few modifications were made, however. Coagulation work was done with a Hach Floc Tester, essentially a gang stirrer, which consists of six magnetic stirrers connected in tandem and set to operate at 60 rpm. Coagulants were added with a dasher mixer, and mixing time was 30 seconds. The slow mix time was 15 minutes at 60 rpm.

Ivory Soap Based Wash Water

A synthetic Ivory Soap (sodium stearate) based wash water was prepared according to the following formulation:

Material	ppm
Ivory Soap	1000
Sodium chloride	500
Sodium sulfate	150
Lactic acid	100
Urea	50

The solution was prepared with distilled water. The Ivory Soap was predissolved by heating in a 65°C oven in a portion of the water.

The evaluation criteria were as before.

The first set of experiments was devoted to establishing the optimum dosage of ferric chloride (Table 12); 170-185 ppm of ferric chloride removed 95-97 percent of the sodium stearate. Use of polymeric flocculants (Table 13) in conjunction with the ferric chloride did not enhance the soap removal.

Aluminum sulfate was also evaluated as a primary coagulant (Table 14). Dosages of 235-300 ppm gave promising results. This work will be continued in a follow-on effort.

Igepon CN-42 Based Wash Water

A synthetic wash water was prepared using 1000 ppm (active surfactant) of Igepon CN-42 (GAF Corporation), sodium N-cyclohexyl-N-palmitoyl taurate. Preliminary precipitation experiments using ferric chloride (Table 15) were not promising.

Palmeto Based Wash Water

Rochester Germicide Co. has recently replaced Olive Leaf Soap with a similar product called Palmeto, also a liquid potassium soap. A synthetic wash water (Table 16) was prepared using 1000 ppm (active soap) of Palmeto; preliminary precipitation experiments using ferric chloride revealed that approximately 95 percent of the soap could be removed with a dosage of 175 to 180 ppm ferric chloride.

Palmeto as well as other candidate surfactants will be further evaluated in a follow-on effort.

TABLE 12

Chemical Precipitation with Ivory Soap Based Wash Water
Ferric Chloride Series

Experiment Number	Coagulant - $FeCl_3$ (ppm)	pH		Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)[1]	% Soap Removed
		Initial	Final				
A-2114-A	50	8.5	6.9	0	0	-	-
A-2114-B	100	8.5	6.1	0	0	-	-
A-2114-C	150	8.5	3.8	1	2	-	-
A-2114-J	170	8.5	3.7	5	5	50	95.0
A-2114-L	175	8.5	3.7	5	5	37	96.3
A-2114-M	180	8.5	3.8	5	5	-	-
A-2114-G	185	8.5	3.5	5	5	29	97.1
A-2114-N	190	8.5	3.6	4	2	-	-
A-2114-D	200	8.5	3.1	2	2	-	-
A-2114-H	215	8.5	3.3	1	1	-	-
A-2114-I	230	8.5	3.2	1	1	-	-
A-2114-E	250	8.5	2.9	2	2	-	-
A-2114-K	265	8.5	3.1	2	2	-	-
A-2114-F	300	8.5	2.9	2	2	-	-

(1) By carbon tetrachloride/IR analysis for carbonyl at 1710 cm^{-1}.

TABLE 13

Chemical Precipitation with Ivory Soap Based Wash Water
Ferric Chloride/Reten 425[1] Series

Experiment Number	Coagulant/Flocculant (ppm)		pH		Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)[2]	% Soap Removed
	$FeCl_3$	Reten 425	Initial	Final				
A-2117-A	185	1.00[3]	8.5	3.5	5	1 [5]	36	96.4
A-2117-B	185	0.50[3]	8.5	3.5	5	1 [5]	-	-
A-2117-C	185	0.25[3]	8.5	3.6	5	1 [5]	-	-
A-2117-D	185	0.10[3]	8.5	3.6	5	1 [5]	-	-
A-2117-E	185	1.00[4]	8.5	3.4	5	4	-	-
A-2117-F	185	0.50[4]	8.5	3.4	5	4	112	88.8
A-2117-G	175	1.0 [4]	8.5	3.5	5	3	47	95.3
A-2117-H	175	0.50[4]	8.5	3.5	5	3	25	97.5

(1) High molecular weight anionic polyacrylamide (Hercules).

(2) By carbon tetrachloride/ IR analysis for carbonyl at 1710 cm^{-1}.

(3) Reten 425 added simultaneously with ferric chloride.

(4) Reten 425 added 5 minutes after ferric chloride.

(5) Floc floats.

TABLE 14

Chemical Precipitation with Ivory Soap Based Wash Water
Aluminum Sulfate Series

Experiment Number	Coagulant/Flocculant (ppm)		pH		Flocculation Size and Rate	Settling Rate
	$Al_2(SO_4)_3$	Reten 425	Initial	Final		
A-2118-A	50	-	8.5	8.0	0	0
A-2118-B	100	-	8.5	7.6	1	1
A-2118-C	150	-	8.5	7.2	1	1
A-2118-D	200	-	8.5	6.7	1	2 (1)
A-2118-G	235	-	8.5	5.1	5	2 (1)
A-2118-E	250	-	8.5	4.9	5	2 (1)
A-2118-H	265	-	8.5	4.3	5	2 (1)
A-2118-F	300	-	8.5	4.0	5	2 (1)
A-2119-A	250	1.00	8.5	4.3	5	2 (1)
A-2119-B	250	0.50	8.5	4.3	5	2 (1)
A-2119-C	250	0.25	8.5	4.4	5	2 (1)
A-2119-D	250	0.10	8.5	4.3	5	2 (1)

(1) Floc floats

TABLE 15

Chemical Precipitation with Igepon CN-42 Based Wash Water
Ferric Chloride Series

Experiment Number	Coagulant/ $FeCl_3$ (ppm)	pH Initial	pH Final	Flocculation Size and Rate	Settling Rate
A-2120-A	50	7.0	4.8	0	0
A-2120-B	100	7.0	3.5	0	0
A-2120-C	150	7.0	3.3	0	0
A-2120-D	200	7.0	3.1	0	0
A-2120-E	250	7.0	3.0	0	0
A-2120-F	300	7.0	2.8	0	0
A-2120-G	350	7.0	2.8	0	0
A-2120-H	400	7.0	2.8	2	2
A-2120-I	450	7.0	2.7	2	2
A-2120-J	150	2.7	2.5	0	0
A-2120-K	200	2.7	2.5	0	0
A-2120-L	250	2.7	2.5	0	0
A-2120-M	300	2.7	2.4	2	2
A-2120-N	350	2.7	2.4	4	2
A-2120-O	400	2.7	2.4	4	2

TABLE 16

Palmeto Soap Based Wash Water
Ferric Chloride Series

Experiment Number	Ferric Chloride Dosage (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal
A-3336-1	150	2	3	-	-
A-3336-2	160	3	3	-	-
A-3337-7	170	4	5	107	89.3
A-3337-8	175	4	5	46	95.4
A-3337-9	180	4	5	51	94.9
A-3337-10	185	4	5	83	91.7
A-3337-11	190	4	5	-	-
A-3337-12	195	4	5	-	-
A-3336-6	210	5	4	-	-

VI. PRELIMINARY DETERMINATIONS ON THE EFFECT OF ANTIFOAMS ON SOAP REMOVAL

There was some concern at the beginning of the program that the use of antifoam agent with wash water would interfere with chemical precipitation of the soap. To determine the effect, if any, we mixed promising antifoams with synthetic wash water, ran the chemical precipitation and analyzed for residual soap.

Our initial efforts were with Olive Leaf Soap based wash water. In each set of experiments, the antifoam was used at its minimum effective dosage (i.e. DB-110 at 100 ppm, Anti-foam 71 at 100 ppm, etc.). Chemical precipitation was performed as before(1).

The first set of precipitations was done with an Olive Leaf Soap based wash water containing 100 ppm of DB-110 silicone antifoam (Table 17). Although the required dosage of ferric chloride was somewhat greater than normal (190 to 200 ppm versus 170 to 180 ppm), the degree of soap removal was still very high, 95 to 98 percent.

In the second set of coagulation studies, we used wash water containing 100 ppm of Antifoam 71, another silicone material. Again we achieved a high level of soap removal despite the antifoam, but in this case there was no effect on the required dosage of ferric chloride (Table 18).

In the next three sets of coagulation experiments, we used heavy metal ions as the antifoam agent. The ions precipitate a portion of the soap to form metal palmitates and stearates which act as an antifoam. For this reason, the required level of ferric chloride necessary for precipitation was altered in each set of experiments (Tables 19, 20 and 21).

(1) First Quarterly Report to NASA-JSC under Contract NAS 9-14518, May, 1975, and Final Report to NASA-JSC under Contract NAS 9-13536, July, 1974

For wash water containing ferric chloride as the antifoam, chemical precipitation can be accomplished with 60 ppm additional ferric chloride (Table 19). Wash water containing aluminum sulfate as the antifoam was effectively treated with 110 to 160 ppm ferric chloride (Table 20). Wash water containing magnesium sulfate as the antifoam was successfully pre-treated with 160 ppm ferric chloride (Table 21).

One additional silicone was also evaluated. Precipitation was run (Table 22) using an olive leaf soap based wash water containing 150 ppm active AF-75 solids. Although the dosage of ferric chloride necessary to coagulate the soap was somewhat greater than normal (200 ppm versus 170-180 ppm), the degree of soap removal was still quite high, 94%, despite the antifoam.

From the above experiments it can generally be concluded that the anti-foams which are effective for Olive Leaf Soap based wash water have no adverse effect on the soap removal.

The next two sets of experiments used Ivory Soap as the cleaning agent (Tables 23 and 24). When DB-110 was used as the antifoam, there was no increase in the required dosage of ferric chloride coagulant and no adverse effect on removal. A dosage of 190 ppm ferric chloride removed 99% of the soap (Table 22).

With Antifoam 71 as the antifoam agent, only 160-180 ppm of ferric chloride was necessary to remove 96% of the soap (Table 24).

The final three sets of experiments used Palmeto as the cleaning agent (Tables 25, 26 and 27).

When DB-110 was used as the antifoam, there was a slight increase in the required dosage of ferric chloride coagulant and no adverse effect on removal (Table 26).

When SWS-211 antifoam was used as the antifoam, there was a significant increase in the required dosage of ferric chloride although, again, there was no adverse effect on removal. A dosage of 210 ppm ferric chloride removed an apparent 99 percent of the soap (Table 27).

For wash water containing ferric chloride as the antifoam, chemical pre-cipitation can be accomplished with 80 ppm additional ferric chloride (Table 25).

TABLE 17

Effect of DB-110 Antifoam[1] on Chemical Precipitation Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal
A-3321-1	160	4	4	-	-
A-3321-2	170	4	4	-	-
A-3321-3	180	4	4	-	-
A-3321-4	190	4	4	13	98.7
A-3321-5	200	4	4	55	94.5
A-3321-6	210	4	4	45	95.5

(1) 100 ppm of DB-110 mixed into wash water prior to chemical precipitation experiments.

TABLE 18

Effect of Antifoam 71(1) on Chemical Precipitation Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant / Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal
A-3321-7	160	4	4	48	95.2
A-3321-8	170	4	4	16	98.4
A-3321-9	180	4	4	41	95.9
A-3321-10	190	4	4	-	-
A-3321-11	200	4	4	-	-
A-3321-12	210	4	4	-	-

(1) 100 ppm Antifoam 71 added to the wash water prior to chemical precipitation.

TABLE 19

Effect of Ferric Chloride(1) Antifoam on Chemical Precipitation Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Floccu-lation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal	Comments
A-3322-1	60	4	4	59	94.1	-
A-3322-2	70	4	4	72	92.8	-
A-3322-3	80	3	4	-	-	Cloudy
A-3322-4	90	2	4	-	-	Cloudy
A-3322-5	100	1	4	-	-	Cloudy
A-3322-6	110	0	0	-	-	-

(1) 100 ppm of Ferric Chloride added to the wash water prior to chemical precipitation.

TABLE 20

Effect of Aluminum Sulfate(1) Antifoam on Chemical Precipitation Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Floccu-lation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal	Comment
A-3324-1	60	0	0	-	-	-
A-3324-2	70	0	0	-	-	-
A-3324-3	80	0	0	-	-	-
A-3324-4	90	0	0	-	-	-
A-3324-5	100	4	4	-	-	-
A-3324-6	110	4	4	41	95.9	-
A-3324-7	120	4	4	14	98.6	-
A-3323-1	130	4	4	58	94.2	-
A-3323-2	140	4	4	-	-	-
A-3323-3	150	4	4	-	-	-
A-3322-7	160	4	4	40	96.0	Slightly Cloudy
A-3322-8	170	4	4	-	-	Slightly Cloudy
A-3322-9	180	4	Cloudy	-	-	-

(1) 100 ppm Aluminum Sulfate added to the wash water prior to chemical precipitation.

TABLE 21

Effect of Magnesium Sulfate(1) on Chemical Precipitation Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	% Soap Removal
A-3324-8	60	0	0	-	-
A-3324-9	70	0	0	-	-
A-3324-10	80	0	0	-	-
A-3324-11	90	0	0	-	-
A-3324-12	100	0	0	-	-
A-3324-13	110	0	0	-	-
A-3324-14	120	3	Cloudy	-	-
A-3324-15	140	4	4	78	92.2
A-3324-16	160	4	3	12	98.8
A-3324-17	180	4	Cloudy	-	-
A-3324-18	200	4	Cloudy	-	-
A-3324-19	220	3	Cloudy	-	-

(1) 100 ppm Magnesium Sulfate added to the wash water prior to chemical precipitation.

TABLE 22

Effect of Antifoam AF-75[1] on Chemical Precipitation Using Olive Leaf Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rates	Actual Soap Content (ppm)	Percent Soap Removed
A-2128-A	160	4	3	-	-
A-2128-B	170	4	4	-	-
A-2128-C	180	4	4	77	92.3
A-2128-D	190	4	4	63	93.7
A-2128-E	200	4	4	58	94.2

(1) 150 ppm active solids

TABLE 23

Effect of DB-110 Antifoam(1) on Chemical Precipitation Using Ivory Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rates	Actual Soap Content (ppm)	Percent Soap Removed
A-3324-20	160	5	3	-	-
A-3324-21	170	5	3	-	-
A-3324-22	180	5	4	-	-
A-3324-23	190	5	4	8	99.2
A-3324-24	200	5	4	29	97.1
A-3324-25	210	5	4	28	97.2

(1) 100 ppm active solids

TABLE 24

Effect of Antifoam 71[1] on Chemical Precipitation Using Ivory Soap Based Wash Water

Experiment Number	Coagulant/ Ferric Chloride (ppm)	Flocculation Size and Rate	Settling Rates	Actual Soap Content (ppm)	Percent Soap Removed
A-3325-1	160	5	4	45	95.5
A-3325-2	170	5	4	34	96.6
A-3325-3	180	5	4	40	96.0
A-3325-4	190	5	4	-	-
A-3325-5	200	5	4	-	-
A-3325-6	210	5	4	-	-

(1) 100 ppm active solids

TABLE 25

Effect of Ferric Chloride [1] as an Antifoam on Chemical Precipitation Using Palmeto Soap Based Wash Water

Experiment Number	Ferric Chloride Dosage (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	Percent Soap Removed
A-3337-13	70	3	4	-	-
A-3337-14	75	4	4	107	89.3
A-3337-15	80	4	4	29	97.1
A-3337-16	85	4	4	-	-

(1) 100 ppm active $FeCl_3$

TABLE 26

Effect of DB-110 (1) Antifoam on Chemical Precipitation of Palmeto Soap Based Wash Water

Experiment Number	Ferric Chloride Dosage (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	Percent Soap Removed
A-3337-17	170	4	4	-	-
A-3337-18	175	4	4	-	-
A-3337-19	180	4	4	-	-
A-3337-20	190	5	4	38	96.2

(1) 250 ppm active solids

TABLE 27

Effect of SWS-211[1] Antifoam on Chemical Precipitation of Palmeto Soap Based Wash Water

Experiment Number	Ferric Chloride Dosage (ppm)	Flocculation Size and Rate	Settling Rate	Actual Soap Content (ppm)	Percent Soap Removed
A-3338-1	170	3	3	-	-
A-3338-2	175	3	3	-	-
A-3338-3	180	3	3	-	-
A-3338-4	190	4	4	267	73.3
A-3338-5	200	4	5	-	-
A-3338-6	210	4	5	5	99.5

(1) 250 ppm active solids

VII. LOW-FOAMING SURFACTANTS FOR SPONGE BATHING AND HAND WASHING

For upcoming missions, there is a need for a personal cleansing agent for sponge bathing and hand washing. This soap should be a "low foamer" and preferably be a liquid. Our approach to the problem was as follows:

(1) Procure recommended low-foaming, nonirritating surfactants from manufacturers.

(2) Evaluate the surfactants for foaming characteristics.

(3) Obtain from custom formulators, low-foaming formulated soaps which incorporate promising surfactants; or prepare such compositions based on surfactant manufacturers' recommendations.

(4) Conduct a thorough evaluation of formulated soaps.

The following low-foaming surfactants were procured:

Surfactant	Manufacturer	Chemical Type	Physical Form	% Active
Pluronic 25 R4	BASF-Wyandotte	Propylene oxide/ethylene oxide/propylene oxide block copolymer.	Paste	100
Pluronic 25 R2	Same	Same	Liquid	100
Pluronic L62	Same	Ethylene oxide/propylene oxide/ethylene oxide block copolymer.	Liquid	100
Pluronic L61	Same	Same	Liquid	100
Pluronic L64	Same	Same	Liquid	100

. . . Continued

Surfactant	Manufacturer	Chemical Type	Physical Form	% Active
Altarox BL-330	GAF Corp.	Nonionic, modified, straight-chain aliphatic polyether.	Liquid	94
Alipal CO-436	GAF Corp.	Sodium salt of a sulfated alkylphenoxy-poly(oxy-ethylene) ethanol.	Liquid	58
Igepon CN-42	GAF Corp.	Sodium N-cyclohexyl-N-palmitoyl-taurine	Paste	23
Triton CF-21	Rohm & Haas	Alkyl aryl polyether	Liquid	100
Triton CF-32	Rohm & Haas	Amine polyglycol condensate	Liquid	95

These surfactants plus controls were evaluated for foaming characteristics according to NASA foaming standard (Recommended Tentative Standards for Wash Water in Manned Spacecraft). Solutions (0.1% active surfactant) were prepared using distilled water, and were tested as follows:

(1) 100 ml of surfactant solution was added to a clean 250 ml graduated cylinder.

(2) The cylinder was shaken for 15 seconds.

(3) The cylinder was placed on the bench and the foam height was measured in centimeters after 15, 30, and 60 seconds. These tests were done at room temperature and also at 30°C and 35°C for promising materials. The results are listed in Table 28.

None of the surfactants gave nonpersistent foams in 15 seconds or less at ambient temperature, as required by the Wash Water Specification. The more promising materials include Triton CF-32, Pluronic 25R4, Pluronic 25R2, Altarox BL-330, Neutrogena, and blends of Pluronics L64 and L61.

Some surfactants, when heated above their cloud points, will exhibit reduced foaming. Note the effect of temperature on Triton CF-32 (cloud point, 25°C), Altarox BL-330 (cloud point, 30°C), Pluronic L62 (cloud point, 32°C), and blends of Pluronics L64/L61. Triton CF-32 and 50/50 Pluronic L64/L61, if used at 35°C, will meet the NASA tentative foaming standard.

The requirement for a low-foaming personal cleansing agent is unique. Soap formulators are attempting the opposite: they want as much foam from their product as possible. None of the presently used soaps (salts of fatty acids such as sodium stearate) will provide a low foam; therefore, little or no work has been done by soap formulators with low-foaming surfactants such as have been investigated above.

The next step should be to contact surfactant manufacturers to determine what work, if any, has been done on formulation of personal hygiene cleansing agents using low-foaming surfactants. It is likely that, using manufacturers' recommendations, such formulations will have to be prepared and evaluated in the laboratory for detergency, foaming, and dermatological characteristics.

TABLE 28

Foam Height[1] Versus Temperature and Time for Various Surfactant Solutions[2]

Surfactant	Foam Height (cm)								
	23°C			30°C			35°C		
	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
Ivory Soap	> 20	> 20	20	-	-	-	-	-	-
(Control)	> 20	> 20	19.5	-	-	-	-	-	-
Olive Leaf Soap	~ 15	11	10	-	-	-	-	-	-
(Control)	~ 13	~ 13	12.5	-	-	-	-	-	-
Miranol JEM	8.0	7.0	6.8	-	-	-	-	-	-
(Control)	9.0	8.0	6.5	-	-	-	-	-	-
Neutrogena	~ 4	~ 4	~ 4	-	-	-	~ 12	~ 9	-
(Control)	3.5	3.5	3.5	-	-	-	7.5	~ 7	-
Pluronic	5.3	1.7	0.5	-	-	-	4.4	3.0	1.4
25R2	5.6	3.2	0.5	-	-	-	-	-	-
Pluronic	5.7	4.5	2.5	-	-	-	2.2	1.4	-
L62	5.2	3.1	1.4	-	-	-	1.3	0.7	-
Triton	1.4	0.2	0	0.6	0.2	0.2	0	0	0
CF-32	1.3	0.2	0	0.3	0.2	0.2	0	0	0
Altarox	4.8	2.2	1.4	1.2	1.2	-	2.1	2.0	1.1
BL-330	3.2	1.6	1.3	2.5	1.2	1.2	-	-	-
Pluronic	4.3	0.2	0	-	-	-	-	-	-
25R4	3.4	0.2	0	-	-	-	-	-	-

(1) NASA foaming standard (Recommended Tentative Standards for Wash Water in Manned Spacecraft).

(2) 0.1% solution in distilled water.

...Continued

Table 28(Continued - 2)

Surfactant	Foam Height (cm)								
	23°C			30°C			35°C		
	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
Alipal CO-436	$>$ 20	-	20	-	-	-	-	-	-
Igepon CN-42	12.5 9.5	- 9	10 7.5	- -	- -	- -	- -	- -	- -
90 Pluronic L64 with 10 Pluronic L61	7.5 6.5	$\sim$6 $\sim$5	4 $\sim$3	- -	- -	- -	- -	- -	- -
50 Pluronic L64 with 50 Pluronic L61	0.9 1.1	0.5 0.7	0.3 0.4	0.7 -	- -	- -	0 -	- -	- -
70 Pluronic L64 with 30 Pluronic L61	1.9	1.0	0.8	2.4	-	0.9	-	-	-

VIII. IN-SITU ANTIFOAMS FOR OLIVE LEAF SOAP

For convenience in defoaming wash water, it would be desirable to be able to synchronize the proper dosage of antifoam with soap usage. For liquid soaps, this could be done by premixing the soap and antifoam in the desired proportions and dispensing them together. To check the feasibility of this concept we conducted experiments to determine:

1. If, when mixed with the soap, the antifoam would continue to function effectively.

2. If the antifoam would form a stable dispersion with the soap.

3. If the antifoam would leave an undesirable greasy residue on the hands.

Our evaluations were done with Olive Leaf Soap (a 21% aqueous solution of a potassium soap) and silicone antifoams DB-110 (Dow Corning Corporation) and Antifoam Emulsion AF-75 (General Electric Company).

To evaluate antifoam effectiveness, we prepared synthetic wash water as usual, but used a soap/antifoam premix. This premix was prepared by shaking the two materials together briefly. Antifoaming was again evaluated by the shake test (Table 29).

Both antifoams were effective at 200 ppm.

Unfortunately, both soap/antifoam dispersions separated on standing, the silicone rising to the surface. We tumbled the mixes on a jar roll for several hours, but the blends still separated on standing.

During the fourth quarter we attempted to compound the antifoams into the Olive Leaf Soap using a ball mill; we also evaluated a finer particle size silicone antifoam, SWS-211 (SWS Silicones Corporation) which we hoped would form a more stable dispersion in the Olive Leaf Soap. In both cases, the mixtures settled into two layers, although the antifoam remained "dispersed". The bottom layers were silicone rich silicone/soap mixtures and the upper layers were all soap. The mixtures were easily re-homogenized with gentle agitation.

It is possible that the settling problem could be overcome with use of a thickening agent in the soap.

TABLE 29

Foaming Character of Wash Water Prepared From Olive Leaf Soap/Antifoam Premix

Antifoam	Residual Foam Height (cm) at Two Antifoam Dosages					
	100 ppm			200 ppm		
	15 sec	30 sec	60 sec	15 sec	30 sec	60 sec
DB-110	0.8 0.8	0.6 0.7	0.5 0.5	0 0	0 0	0 0
AF-75	0.4 0.5	0.3 0.2	0.2 0.2	0 0	0 0	0 0

In order to check "compatibility with man", two technicians in our laboratory washed their hands with the soap/antifoam mixture; neither noticed any residue or greasy feel on their hands. Patch tests are advisable, however, since some individuals are sensitive to silicone.